# BULLETIN

## OF THE INSTITUTE OF METALS

VOLUME 1 SEPTEMBER 1952 PART 13

## INSTITUTE NEWS

### Symposia on "The Control of Quality in the Production of Wrought Non-Ferrous Metals"

The Council has approved a recommendation of the Metallurgical Engineering Committee that a series of three all-day symposia on "The Control of Quality in the Production of Wrought Non-Ferrous Metals" be arranged in connection with the 1953, 1954, and 1955 Annual General Meetings. The three symposia will cover, respectively: melting and casting (1953), working (1954), and heat-treatment and finishing (1955).

The first of the series—for which six papers have been invited—will be held at the Park Lane Hotel, Piccadilly, London, W.1, on Wednesday, 25 March 1953. The papers will be printed in the *Journal* before the meeting, more details of which will be published in a later issue of the *Bulletin*. Will members please note that offers of additional papers for this Symposium cannot be accepted.

### 1953 Annual General Meeting 23 to 26 March 1953

As previously announced, the next Annual General Meeting of the Institute will be held in London from Monday, 23 to Thursday, 26 March 1953, inclusive. The scientific sessions, a luncheon, and a dinner-dance will be held at the Park Lane Hotel, Piccadilly, London, W.1.

The meeting will open on the evening of Monday, 23 March, when the 43rd Annual May Lecture will be delivered. After the business meeting and the Presidential Address on the morning of the following day, there will be a Luncheon at which the Institute's medals will be presented. There will be one or more scientific sessions in the afternoon, and in the evening there will be a dinner-dance.

As announced above, there will be an all-day symposium on Wednesday, 25 March on "The Control of Quality in the Production of Wrought Non-Ferrous Metals. I.—Control of Quality in Melting and Casting", and it is possible that alternative sessions may also be arranged for the discussion of scientific papers. In the evening an informal conversazione will be held at 4 Grosvenor Gardens, S.W.1.

A series of visits to works and laboratories in the London area is being arranged for Thursday, 26 March.

Members are requested to note these dates. It is hoped that the meetings and social functions will be well attended.

### Informal Discussion on "Rolls and Their Maintenance in the Non-Ferrous Metals Industry"

Under arrangements made by the Metallurgical Engineering Committee, an Informal Discussion on "Rolls and Their Maintenance in the Non-Ferrous Metals Industry" will be held at the University, Edgbaston, Birmingham 15, on Thursday, 8 January 1953.

The meeting will take place in the afternoon. By the kind invitation of Professor A. J. Murphy, members and visitors will be able to inspect the laboratories of the Industrial Metallurgy Department of the University in the morning, and those who desire to do so will—provided that they give 10 days' warning—be able to take lunch in the University refectory.

Full particulars of this meeting will be published later.

### Educational Tour for Junior Members and Student Members

An Educational Tour for Junior Members and Student Members is to take place in the Birmingham area from Monday to Friday, inclusive, during a week in the 1953 Easter vacation. Further particulars will be published in due course.

### Symposium on "Properties of Metallic Surfaces", London, 19 November 1952

Members are reminded that an all-day Symposium on "Properties of Metallic Surfaces", arranged by the Metal Physics Committee, will be held at the Royal Institution, Albemarle Street, London, W.1, on Wednesday, 19 November 1952.

Copies of the programme, with Registration Form, were posted to all members at the end of July.

### Joint Meeting in Bristol

A joint meeting between local members of the Institute and members of the Bristol Sections of the Chemical Society, the Royal Institute of Chemistry, and the Society of Chemical Industry will be held in the Chemical Department of the University, on Thursday, 2 October 1952, at 7 p.m.

Major P. L. Teed, A.R.S.M., F.R.Ae.S., will give a lecture on "Some Metallurgical Problems Imposed by Stratospheric Flight".

## Acta Metallurgica : A New International Journal of the Science of Metals

At the World Metallurgical Congress, organized by the American Society for Metals in Detroit, in October 1951, representatives of that Society discussed with a group of interested visitors from overseas a tentative plan for the publication of a new international journal of the science of metals. It was stated that research workers in the field of solid-state physics, and certain chemists and metallurgists, had felt the need for a new scientific periodical that would cover fields which lie beyond the scope of current metallurgical journals and the literature of which is now scattered, often inappropriately, in a diversity of physical and chemical publications. The A.S.M. fostered this movement, and it was reported at the World Congress that its Board of Trustees had appropriated funds for initiating the journal.

After an Interim Board of Governors, appointed by the A.S.M., had considered comments made by various persons regarding the original proposals, it produced the outlines of a scheme for consideration by the more important chemical, metallurgical, and physical societies of the world, which were invited to be associated with the production of the proposed journal, either as "sponsoring" societies (with financial liability) or as "co-operating" societies (without financial liability).

Your Council appointed the late Sir William Griffiths (a Past-President) to be its representative at discussions which took place on this matter in the U.S.A., and the President and immediate Past-President have also had opportunities to discuss the proposals with members of the Interim Board of Governors.

After careful consideration, the invitation to the Institute of Metals to be associated with the publication of this new journal has now been accepted. It has been agreed that the Institute shall be a "co-operating" society. As such it will form one of a group of societies which will be entitled to representation on the Board of Governors. In addition, members of the Institute will be enabled to subscribe to the new periodical at privileged rates, particulars of which will be announced in due course.

For the information of members, a copy of the Institute's reply is printed below.

Dear Dr. Hollomon,

"ACTA METALLURGICA"

The Council of the Institute of Metals has now had an opportunity to meet and discuss the invitation, contained in your letter of 1 April 1952, for the Institute to be associated with the publication of the proposed new international journal of the science of metals, which we learn is to be known as *Acta Metallurgica*. Certain of the Institute's officers have also had conversations with officers of the Iron and Steel Institute on this matter.

When your proposal first came before us we felt some concern as to the effect, if any, that this new publication might have on our own *Journal*. The discussions, however, that our representatives have had on this matter with your Interim Board have proved most encouraging. We appreciate that the field which you propose to cover in *Acta Metallurgica* will mainly include papers which aim to contribute to the understanding of the properties, structure, and behaviour of metals in terms of the fundamental particles, forces, and energies that are involved, and similar fields of metal physics.

Since such papers have in the past formed a negligible proportion of our own publications, we feel that your new journal will not be a mere duplication of existing media of publication but will, in fact, fill a very definite need.

On this understanding, the Council of the Institute of Metals is glad to accept your invitation to the Institute to become a co-operating society. It regrets, however, that the Institute is not in a financial position to become a sponsoring society.

Yours very truly,

*London*,
15 *July* 1952.

(signed) C. J. SMITHELLS *President*
F. C. THOMPSON *Senior Vice-President*
S. C. GUILLAN *Secretary*

## Reduced Rates of Subscription for Members Retired from Active Professional Life

Members who have retired from their main professional occupation, are not less than 65 years of age, and have been members of the Institute for not less than 25 years may claim the privilege of paying a reduced rate of annual subscription, on application to the Secretary for details regarding this concession, which has recently been approved by the Council.

## Binding of the Bulletin

Volume I of the *Bulletin* will continue until the August 1953 issue, when a title page and index will be published to enable this publication to be bound, for permanent record.

Members who, in due course, will send their monthly issues for binding, should preserve the pages of the *Bulletin* until the first volume is completed. It is expected that in future years the volume of matter printed in the *Bulletin* will be enough to justify binding the periodical annually, at the same time as the *Journal* and *Metallurgical Abstracts*.

## Binding of Plates in the Journal

It has been decided to continue, until the August 1953 issue of the *Journal*, the experiment of binding plates in one place in each issue, instead of within the individual papers to which they refer. The practice has effected a useful financial saving.

In future the plates will be inserted at the end of the *Journal* section of each monthly issue.

## Deaths

The Editor regrets to announce the death of:

MR. ISRAEL JULES ESPIR, Director of Fernand Espir, Ltd., Northwood, Middlesex, on 31 May 1952.

MR. WILLIAM ROBERT FRANKLIN, Ministry of Supply, in the Middlesex Hospital, London, on 24 June 1952. He had been a member of the Institute since 1919.

SIR WILLIAM THOMAS GRIFFITHS, D.Sc., in hospital at Redhill, Surrey, on 29 July, aged 57. Sir William, who was formerly Chairman and Managing Director of The Mond Nickel Company, Ltd., and a Vice-President of the International Nickel Company of Canada, served as President of the Institute of Metals in 1944–46. The Institute was represented at his funeral by Dr. L. B. Pfeil (Member of Council).

## Election of Members

The following 18 Ordinary Members, 2 Junior Members, and 6 Student Members were elected on 25 July 1952:

*As Ordinary Members*

ATKINSON, Ralph W., B.Sc., E.E., Director of Research, General Cable Corp., 246 First Street, Bayonne, N.J., U.S.A.

BARTEN, Ernst H., Dr.-Ing., Works Superintendent and Partner, Achenbach Söhne G.m.b.H., Siegenerstr., Buschhütten (Kr. Siegen), Germany.

BATCHELOR, Leslie Charles, A.I.M., Departmental Manager, Refinery and Casting Departments, Imperial Chemical Industries, Ltd., Metals Division, Elliott Works, Selly Oak, Birmingham 29.

BRYANT, Anthony James, Assistant Metallurgist, Plant Control Department, Northern Aluminium Co., Ltd., Banbury, Oxon.

ERDMANN-JESNITZER, *Professor* Friedrich, Dr.-Ing.habil., Professor of Metallurgy, Institut für Metallkunde der Bergakademie, Freiberg (Sachsen), Germany.

GATTONI, Attilio, Dr.-Ing., Managing Director, Fabrica Italiana Tubi Metallici S.p.A., Corso Francia N.252, Torino, Italy.

GILBERT, Edward Albert, Chief Chemist and Metallurgist, T. J. Priestman, Ltd., Leopold Street, Birmingham.

LÖHBERG, Karl August, Dr.phil.habil., Deputy Chief, Metall-Laboratoriums der Metallgesellschaft A.G., Frankfurt-am-Main, Germany.

LOVE, Ralph Edward, Chemist, Mono Pumps, Ltd., 67 Clerkenwell Road, London, E.C.1.

MEHTA, M. L., B.Sc. (Met.), Research Student, Department of Metallurgy, University of Manchester.

POPOFF, Anatole, Ing.-Dr., Research Engineer, Office National d'Etudes et de Recherches Aéronautiques, Chatillon-sous-Bagneux (Seine), France.

RICE, John Leslie, B.Met., British Non-Ferrous Metals Research Association, Euston Street, London, N.W.1.

SALMON, Guy, Research Engineer, Fonderies et Forges de Crans, Cran-Gevrier, Annecy (Haute-Savoie), France.

SPIRYTUS, Jacques, Ing. E.S.T.D., Directeur-Général, S.A. Affinerie de Juvisy, 31 Quai de l'Industrie, Juvisy (S. et O.), France.

STAGNI, Otello C., Dr.-Ing., Ing. Stagni & C. S.p.A., via Manzoni 41, Milano, Italy.

THOMÉ, Paul, Ing., Research Engineer, Office National d'Etudes et de Recherches Aéronautiques, Chatillon-sous-Bagneux (Seine), France.

THOMSON, Robert F., M.S., Ph.D., Head, Metallurgy Department, Research Laboratories Division, General Motors Corp., P.O. Box 188, North End Station, Detroit 2, Mich., U.S.A.

THORNTON, Ben, Director, The Wellman Smith Owen Engineering Corp., Ltd., Parnell House, Wilton Road, London, S.W.1.

*As Junior Members*

LOMER, W. M., B.A., M.Sc., Ph.D., Theoretical Physics Division, Atomic Energy Research Establishment, Harwell, Didcot, Berks.

MEADOWCROFT, R., B.Sc., Director, Mitchell and Meadowcroft, Ltd., Stonebridge Chambers, Colne, Lancs.

*As Student Members*

ARGENT, Bernard Barry, Student of Metallurgy, University of Sheffield.

BROMFIELD, Gerald, Assistant Metallurgist, The General Electric Co., Ltd., Copsewood, Coventry.

FIELDS, Maurice, Assistant Metallurgist, Alfred Herbert, Ltd., Edgwick, Coventry.

GOREHAM, Alan Richard, Laboratory Assistant, British Non-Ferrous Metals Research Association, Euston Street, London, N.W.1.

JEE, Akshoy Kumar, Student of Metallurgy, c/o Dr. Harnath Jee, Buddhi-thakurani Lane, P. O. Cuttack 2, Orissa, India.

PICKUP, Geoffrey Arthur, B.Met., On National Service.

The following 9 Ordinary Members, 1 Junior Member, and 4 Student Members were elected on 12 August 1952:

*As Ordinary Members*

BISCHOF, Wilhelm, Dr.Ing.habil., Leitender Regierungsdirektor, Staatliches Materialprüfungsamt Nordrhein-Westfalen, Marsbruchstrasse 186, Dortmund-Aplerbeck, Germany.

DUNN, Kenneth George, Technical Officer, Technical Training College, Department of Officer Training, Indian Air Force, Jalahalli, India.

FISHER, Philip Andrew, A.C.T. (Birm.), A.I.M., Metallurgist, Magnesium Elektron, Ltd., Clifton Junction, near Manchester.

HARPER, George Cedric, Senior Technical Assistant, British Oxygen Co., Ltd., North Circular Road, Cricklewood, London, N.W.2.

JAMES, Percy, Aluminium Wire and Cable Co., Ltd., Port Tennant, Swansea.

LECLAIRE, Alan Delamare, B.A., Senior Scientific Officer, Metallurgy Division, Atomic Energy Research Establishment, Harwell, Didcot, Berks.

PHILLIPS, Charles W., M.S., Instructor, Department of Chemical and Metallurgical Engineering, University of Michigan, Ann Arbor, Mich., U.S.A.

SINNOTT, Maurice J., Sc.D., Associate Professor, Department of Chemical and Metallurgical Engineering, University of Michigan, Ann Arbor, Mich., U.S.A.

STEPHENSON, Joseph, B.Met., A.I.M., Research Metallurgist, Metallurgy Division, Atomic Energy Research Establishment, Harwell, Didcot, Berks.

*As Junior Member*

WALKIDEN, George William, B.Sc., Corrosion Research Chemist, The British Aluminium Co., Ltd., Chalfont Park, Gerrards Cross, Bucks.

*As Student Members*

COWEN, Henry Chapman, M.Sc., Assistant Lecturer, Department of Metallurgy, University of Manchester.

CRISP, John, Student of Metallurgy, University of Birmingham.

MILLS, Brian Edward, Laboratory Assistant, British Non-Ferrous Metals Research Association, Euston Street, London, N.W.1.

SMITH, Anthony Aston, B.A., Research Student, Department of Metallurgy, University of Cambridge.

*Student's Essay*

# THE METALLOGRAPHIC INVESTIGATION OF FAILED HIGH-TEMPERATURE COMPONENTS *

By J. C. WRIGHT,† Student Member

*SYNOPSIS : The metallographer engaged in the examination of components which have failed during service at high temperatures, is confronted with a number of specialized problems. Modifications in the technique of preparation and examination of specimens are suggested, and the most common types of failure are reviewed from the point of view of their metallographic implications. It is pointed out that no satisfactory classification of failures is possible, since the mechanism of breakdown is usually complex.*

## Introduction

Metallography may be used in three sections of the metallurgical industry: industrial research, process control, and examination of failures. There is considerable overlapping of the three sections, and there are few metallographers whose duties confine them strictly to one specialized field. Nevertheless, experience of one particular application generally influences the outlook and technique of the metallographer beyond the experience gained in his other duties. The metallographer who deals with failures of refractory alloys demonstrates this ideally. While he must necessarily have a knowledge of the influence of conditions at normal temperatures, his mind is generally preoccupied with the problems associated with high temperatures. The attendant phenomena of creep, oxidation, dry corrosion, and other accelerated structural changes tend to be of greater interest than faults associated with normal temperatures. It is the purpose of this essay to indicate the problems with which the metallographer, engaged in the examination of high-temperature components, is confronted.

An engineering component used at high temperatures is regarded as satisfactory if it gives useful service for more than a certain minimum life. At the end of this time it may collapse or have deformed sufficiently to be rendered useless, but if it has fulfilled the conditions considered in its original design, this can hardly be termed a failure. True failures are due to a component being "below specification", or to its being subject to adverse conditions not taken into account in the design. Most of the failures can be investigated and confirmed satisfactorily by the application of metallography as the main instrument.

## Metallographic Preparation

Although it is not claimed that a revolutionary technique is required for this type of examination, the likelihood of encountering such features as oxide films, cracks, distortion, and intergranular penetration, is sufficient to influence the metallographer in his method of preparation. When selecting portions for microscopical examination, it is advisable to obtain sections through parts of the component which have been relatively unaffected by the heat and destructive conditions, as well as to take sections from the damaged areas themselves. In this way some idea of the state of the component on entering service may be gained, and any subsequent changes from this state more easily noticed. Most specimens require mounting before polishing, and it is advisable to use a transparent plastic for this purpose because this allows the operator to see exactly where the final section has been taken, and also because it is virtually structureless when viewed under the microscope. It is frequently difficult to distinguish between scale on the outside of a specimen and the mounting material, if this is of the black Bakelite type.

While the more common techniques of polishing can be satisfactory in experienced hands, highly successful results can be obtained by less-skilled operators in a much shorter time by using diamond-dust polishing after the grinding process. Standard polishing apparatus may be used, as outlined by Perryman,[1] and the time saved in producing a satisfactory polish makes it an economical proposition. The diamond dust has the advantage in this type of work that it cuts equally well through layers having widely different hardnesses. Thus, a very flat section is produced, and oxide films, carbides, other inclusions, and even very fine cracks are well delineated. A 0–1-$\mu$ diamond-dust polish is satisfactory for ordinary examination at magnifications up to $\times 300$, but above this, very fine scratches can be seen. It has been suggested that the scratches are due to agglomerates of diamond dust of the order of 4 $\mu$ in dia., which are formed as a result of the very high surface energies of the original 1-$\mu$ particles. The scratches can be quickly removed by a light polish on a fine alumina or magnesia pad, but this treatment should be as short and light as possible, or the overall flatness of the surface will be lost. Electrolytic polishing, as developed up to the present, is unsatisfactory for preparing specimens of failures, because the cracks, voids, and irregularities, which are of great interest, are not well reproduced by this method.

Having obtained satisfactory polished and etched microstructures of the failed component, it is the metallographer's job to correlate his microscopical examination with any other observations and data available, and to determine as nearly as possible the cause and method of failure. It is proposed to review the more common types of failure from the aspect of the metallographic observations associated with them, and then to show why no rigid classification of the symptoms of failure can be attempted.

## Mechanical Breakdown

The most common failures under severe mechanical conditions are due to creep or fatigue. According to Sully,[2]

* Awarded the Students' Essay Prize of the Institute of Metals for 1952.

† Laboratory Assistant, Development and Research Department, The Mond Nickel Co., Ltd., Birmingham.

the type of creep which has taken place in a component can be correlated with the type of fracture that occurs. If relatively fast straining has produced distortion at relatively low temperatures, the conditions are similar to those involved in a normal tensile test. The fracture will be transcrystalline and will be accompanied by some local elongation and hence crystal distortion. The more normal type of failure results from a slow rate of straining at high temperatures, and this shows itself by a brittle intercrystalline fracture. The intercrystalline fissures are not confined to the fracture alone, and it is usual to find an area on both sides of the fracture in which the grains have become partially separated. A complete range of fractures from the transcrystalline to the intercrystalline type is formed by failures under intermediate conditions, and it is not unusual to find a crack developing across and between crystals in the same specimen. The distinction between trans- and inter-crystalline creep failure is determined by the difference between the resistance to flow of the crystalline material and of the grain boundary. At high strain rates and short times, the crystalline flow is more pronounced than the more viscous grain-boundary flow, and consequently slip, followed by transcrystalline cracking, is prevalent. Under low strain rates and longer times, grain-boundary flow is more noticeable and the crystals to some extent slide over each other. The stress concentration at a junction builds up until voids appear in the grain boundary and spread to cause an intercrystalline failure. It seems likely that the reason for coarse-grained material having a superior creep-resistance at high temperatures, is that a relatively smaller proportion of grain-boundary material is present to aid intercrystalline flow. Localization of a second constituent or impurity in the grain boundary will also modify creep at high temperatures. It may help to resist intergranular movement, but it is much more likely to provide a source of weakness and lead to the premature appearance of voids.

Failure by fatigue is somewhat different, in that the temperature does not greatly influence the type of fracture produced. Even with a ductile metal a fatigue failure exhibits negligible ductility in the region of the true fatigue crack, though when a section has been reduced by the propagation of such a crack, final separation usually occurs by ordinary rupture. This part of the fracture may be ductile and helps to give the dual-zone appearance characteristic of fatigue failure. The part of the fracture due to the fatigue crack is generally smooth, but may be oxidized if the component was working at high temperature. It frequently shows in addition concentric markings which delineate the progress of the crack, having as the centre a nucleus or stress-raiser zone which often has a more crystalline face.

According to Edwards,[3] a combination of high-temperature creep with fatigue set up by aerodynamic flutter is the chief cause of failure of the high-speed rotary parts of a jet engine, particularly the turbine blades. Mechanically induced vibration is low owing to the absence of reciprocating parts.

The only other cause of failure of a purely mechanical nature which is connected with high temperatures is that due to thermal shock. Components which are repeatedly strongly heated and drastically cooled, frequently start to crack at weak points. The rapid heating and cooling set up thermal gradients in the components, and the variations in expansion or contraction, across a section, lead to high internal stresses. Ageing of a component during this time results in an increased resistance to thermal shock, and components which are in the aged condition at the beginning of their service generally have better thermal-shock properties.[4] The true mode of fracture for pure thermal shock appears to be transcrystalline and at right angles to the main stress.[4, 5] Bentele and Lowthian[6] investigated the effect of thermal shock on the life of gas-turbine nozzle blades and found that the cracks which developed in a thick trailing edge of the blade were truly transcrystalline, whereas those in a thin leading edge were intercrystalline, particularly those near the blade surface. Bentele and Lowthian did, however, use *aqua regia* to remove surface deposits to facilitate crack detection. The disturbing influence of the acid and atmospheric attacks, particularly on thin sections, may be the explanation for their finding occasional intercrystalline fractures. The main thermal-shock crack in a blade, or at the edge of holes punched in flame-tube assemblies, is often surrounded by other cracks. Some originate at the edge, or at least join up with the edge, but often discontinuous cracks are found which appear to originate away from the edge and propagate only a short way before being stopped. Jaffe, Reed, and Mann[7] believe that these discontinuous cracks occur close to all brittle transcrystalline fractures and have observed them microscopically in failed fatigue specimens, impact specimens, and failed components. They have attempted an explanation, which involves the conception that brittle transcrystalline fracture does not originate at one point and propagate continuously across a section, but nucleates at numerous points, leading to a series of microcracks which subsequently join up. The grain-size of high-temperature components frequently alters during service, but as far as is known at present the grain-size does not have much effect on thermal-shock resistance.[5] Small pieces of the matrix are often observed in a thermal-shock crack, completely free from the parent grain. If electrolytic etching is used, there is a good chance of these pieces remaining unetched. The mechanism of their separation is probably complex, but might well involve rubbing between the sides of the crack, combined with oxidation.

## Formation of Unsatisfactory Microstructures during Service

Apart from the physical causes mentioned so far, there is another class of failure which may be attributed to a change in the microstructure of a component, from a satisfactory to a less satisfactory form. The important cases of this kind concern excessive grain growth, spheroidization, and to a lesser extent graphitization. The investigation of this type of failure lends itself particularly to metallographic treatment. Instances of grain growth in service are typified by the reports of Allsop and Bygate[8] and of Hodgson and Baron[9] concerning certain silicon–chromium valve steels. It was noticed that frequent failures were associated with coarse-grained zones which had developed in the head and neck of certain valves during service. The grain growth appeared to be due to the susceptibility of these valve steels to cold work while still at bright-red heat, as a result of which critical strain could remain after working. If, during heating the formed valve for final hardening, austenitization was not complete (and early specifications made this possible), grain growth would occur. Under service conditions, the growth may at times continue, and the unsatisfactory structure produced is notch-brittle at the elevated temperatures commonly experienced by exhaust valves.

Failures due to graphitization are comparatively rare, but it has been shown[10] that carbon and molybdenum steels which have been deoxidized with large amounts of aluminium

are susceptible to graphitization when held for long periods at high temperatures. The planes of weakness are self-evident in the micro-examination of graphitized samples, and in fact formation of weak sections consisting almost wholly of graphite have been reported.[11]

Spheroidization is a rather more common occurrence. Weakness due to the spheroidization of pearlitic steel or to the agglomeration of an already spheroidized structure, is a natural outcome of long periods at elevated temperatures. Deformation of a component at high temperature appears to accelerate the spheroidization process,[12, 13] and this acceleration can be observed in specimens which have failed by creep. Many refractory alloys which do not normally exhibit carbides may show carbide precipitation at working temperatures. This is particularly true of the austenitic stainless type of alloy, but embrittlement resulting from the precipitation is not usually sufficient to cause a breakdown, unless other service conditions, such as dry corrosive action, are severe. It is common practice to stabilize the structure of such alloys by adding a small percentage of titanium or niobium. Any carbon present combines preferentially with these instead of with the chromium. Carbide precipitation does not then denude the matrix of the chromium, and it is thus left in a corrosion-resistant condition. Examination may, however, occasionally reveal such heavy carburization that the stabilizers have become saturated with carbon and some chromium carbide has been precipitated as well. This extreme has been observed in the exhaust manifolds of reciprocating aircraft engines, where carburization has been due to incompletely burnt and very hot exhaust gases.

A section on precipitation of carbide and subsequent oxidation of the matrix would be incomplete without a more detailed description of the outstanding example "green rot". As described by Bucknall and Price,[14] the symptoms of green rot are intergranular oxidation; formation of oxide patches within the grains; a general swelling of the section; and ultimate breakage with a typical greenish fracture. There is also an increase in magnetic response. The failures occur when nickel-chromium alloys are subjected to high temperatures in carburizing atmospheres. A useful example is that of a nickel-chromium electric-furnace heating element, in use in a furnace handling oily components or in a controlled carburizing atmosphere. Micro-examination of failures by green rot shows that carburization of the chromium takes place with consequent swelling, and this is followed by progressive oxidation of the precipitated carbide. Preferential precipitation of carbide in the grain boundaries and its subsequent oxidation presumably accounts for the intergranular fracture, but, as mentioned above, a similar precipitation–oxidation process, without the subsequent fracture, takes place in the grains themselves. It has been noticed that sulphur inhibits the process of green rot, and sulphide masses, if present, appear to move ahead of the oxidation front but slow down its forward movement. Green rot is also opposed by silicon or iron in the alloys.

## Dry-Corrosion Failures

The mention of dry corrosion and its effect on unstable structures opens up another large field of possible causes of failure. Metallographic examination is again the most satisfactory method for determining the exact cause of the trouble. Dry-corrosion mechanisms vary considerably, and in some cases are not well understood. In general there are two main classes. By far the most common is oxidation, the other class being concerned with penetration of miscellaneous weakening materials, such as sulphides, solders, brazing alloys, and fluxes. It is obvious that components designed for high-temperature service should be resistant to oxidation under their normal working conditions, but unfortunately, several compounds commonly met with accelerate the oxidation process. For illustration, these compounds may be represented by the oxides of molybdenum, lead, and vanadium.

Refractory alloys subjected to the influence of molybdic oxide vapour at high temperatures in a quiescent atmosphere, suffer from catastrophic oxidation.[15] The surface of any affected part becomes badly broken up and swells markedly. General, rather than intergranular, oxidation occurs at the metal/metal oxide interface. The oxide formed is very porous and consequently allows free diffusion, thus making the process cumulative. Just ahead of the advancing oxide front it is common to observe a zone of heavy carburization. This is probably due to the fact that carbides diffuse but slowly into the oxide film, and accumulate ahead of the oxidation front. The essential factor in the attack is the lingering presence of molybdic oxide vapour which dissociates rapidly above about 800° C. This reversible dissociation seems to catalyse the oxidation reaction of the atmosphere. In the initial stages of such an attack there is always the possibility that the molybdic oxide, even in liquid form, may destroy protective coatings, such as chromic oxide on chromium steels.[16] There is evidence to support the argument that such a mechanism is the main means by which lead and vanadium oxides promote oxidation corrosion. No intergranular penetration of vanadium oxide has been reported so far. The presence of lead oxide in anti-knock fuel ashes and of vanadium pentoxide in some burnt Diesel-oil ashes make this type of attack quite probable. Metallography usually shows a disturbance of the surface skin and consequent sub-surface oxidation without allowing the initial agent to be identified.

One very common process of dry corrosion met with in refractory alloys, particularly those containing nickel, is that of "sulphur attack". This can usually be satisfactorily explored by microscopical means. The presence of sulphur dioxide or hydrogen sulphide in burnt fuel gases lowers the temperature at which the protective oxide layer on the surface of refractory alloys breaks down. In this way, sulphur attack is achieved by a mechanism similar to that described for the metallic oxides above. The attack is more marked in reducing than in oxidizing conditions. Alloys containing nickel suffer very badly in atmospheres of high sulphur content. This is believed to be due to the formation in the scale of mixtures of nickel sulphide, nickel oxide, and metallic nickel, these mixtures having a possible melting point as low as 650° C. Not only is the protective power of the oxide skin rendered useless, but in the liquid condition of the scale mixture, intergranular penetration is facilitated. Sulphides are fairly easy to identify microscopically by their generally rounded form and greyish colour. If doubt arises, there is a confirmatory test[17] which can be applied in conjunction with observation with a low-power microscope. A mixed solution of sodium azide and iodine liberates tiny bubbles of nitrogen when in contact with sulphides. Care must be taken to see that the solution does not enter cracks or holes and by displacing air give the impression that the sulphide reaction has occurred.

As in the case of attack in atmospheres rich in molybdic oxide, carburization ahead of the advancing corrosion zone is sometimes observed with attack in sulphurized atmospheres. The most common appearance of a section attacked by sulphur

may be described as having wart-like excrescences of corrosion products, beneath which are saucer-shaped depressions in the surface of the original metal. Frequently, the corroded zone shows two different layers. The outer layer is fairly light in colour and is broken, and beneath this a more adherent layer of corrosion products occurs.

Provided careful preparation of suitable microsections is undertaken, as outlined earlier, sources of intergranular corrosion can generally be traced and the corrosive medium, if it be flux, solder, brazing metal, or other low-melting-point material, can usually be identified. It is useful to remember that components which are satisfactory under the influence of such corrosive media under negligible stress conditions, may suffer rapid intergranular attack if stressed in tension.

Another type of intergranular failure met with in components which have been in high-temperature service, is that of incipient fusion at grain boundaries in areas where the material has been locally overheated. Micro-examination of the remaining grains generally indicates a structure typical of a solution-treated condition, although the component may exhibit an aged or some other serviceable structure, at points which have not received such extreme heating. If hardness surveys are carried out on a typical section suffering from this type of fault, it will generally be found that failure has occurred at the softest spot.

### Conclusion

Thus, it will be seen that the mechanisms by which components may break down under high-temperature conditions, may be classified very loosely under three headings: (i) mechanical breakdown, (ii) development of unsatisfactory microstructure, and (iii) dry corrosion. Unfortunately, there are not enough features common to all the variations of failure, which may be grouped under any one of these three main headings, to make classification worthwhile. More important than this is the fact that very rarely in practice does a component fail purely and simply by one single process. It is usual to find one process predominant and others helping the breakdown in a subsidiary way. For instance, thermal-shock cracks generally show oxidation of the fractured surface. Sometimes the oxide layer extends into the metal beyond the visible end of the crack, and it appears that oxidation has assisted the thermal-shock conditions in opening up the void. Frequently it is difficult to decide which particular process started the decline of the component, and which process later helped to accelerate the decline. In most cases, metallographic examination can at least indicate where the trouble started, and usually the mechanism of propagation of the fault can be identified.

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REFERENCES

1. E. C. W. Perryman, *J. Inst. Metals*, 1950, **77**, 61.
2. A. H. Sully, "Metallic Creep and Creep-Resistant Alloys". **1949**: London (Butterworths Scientific Publications).
3. A. R. Edwards, *Symposium on the Failure of Metals by Fatigue, Univ. Melbourne*, **1946**, 383.
4. G. M. Ault and G. C. Deutsch, *S.A.E. Journal*, 1950, **58**, (5), 40.
5. M. J. Whitman, R. W. Hall, and C. Yaker, [*U.S.*] *Nat. Advis. Cttee. Aeronautics, Tech. Note*, 1950, (**2037**).
6. M. Bentele and C. S. Lowthian, *Aircraft Eng.*, 1952, **24**, 32.
7. L. D. Jaffe, E. L. Reed, and H. C. Mann, *Trans. Amer. Inst. Min. Met. Eng.*, 1949, **185**, 526.
8. H. Allsop and P. W. Bygate, *J. Iron Steel Inst.*, 1949, **161**, 318.
9. C. C. Hodgson and H. G. Baron, *ibid.*, 1949, **161**, 81.
10. G. V. Smith, *Weld. J.*, 1948, **27**, 277s.
11. R. W. Emerson and M. Morrow, *Trans. Amer. Soc. Mech. Eng.*, 1946, **68**, 597.
12. C. H. M. Jenkins, G. A. Mellor, and E. A. Jenkinson, *J. Iron Steel Inst.*, 1942, **145**, 51P.
13. S. H. Weaver, *Proc. Amer. Soc. Test. Mat.*, 1941, **41**, 608.
14. E. H. Bucknall and L. E. Price, *Rev. Mét.*, 1948, **45**, 129.
15. W. C. Leslie and M. G. Fontana, *Trans. Amer. Soc. Metals*, 1949. **41**, 1213.
16. G. W. Rathenau and J. L. Meijering, *Metallurgia*, 1950, **42**, 167.
17. F. Feigl, "Qualitative Analysis by Spot Tests". **1947**: New York (Elsevier Publishing Co.).

# OTHER NEWS

## Symposium on Industrial Failure of Engineering Metals and Alloys

A Symposium under this title is to be held at the National Metallurgical Laboratory, Jamshedpur, India, at a date that will be announced later.

The symposium will be broadly divided into two main parts, viz. (i) theoretical aspects of the industrial failure of engineering metals and alloys, and (ii) a study of actual service failures, including an investigation into their causes and possible remedial measures. Technical papers on these subjects detailing, wherever possible, actual service failures, will be greatly welcomed. Those wishing to present papers should forward an advance copy to the organizers, at the National Metallurgical Laboratory, before 31 October 1952, so that the final programme, abstracts of the papers, &c., may be drawn up and circulated.

# APPOINTMENTS VACANT

**A COMPANY** 20 miles west of London requires an assistant chemist for the analysis of copper- and nickel-base alloys, stainless steel. Knowledge of spectrographic analysis an advantage. Salary according to age, qualifications, and experience. Write Box V.248, Willing's, 362 Grays Inn Road, London, W.C.1.

**AN ENGINEER** with a metallurgical background is required for the materials section of the Nelson Research Laboratories, English Electric Company, Ltd., Stafford, for work on the technical control of the processes of melting alloys by high frequency, and hot and cold rolling of strip. Applicants should possess H.N.C. or equivalent and be familiar with rolling-mill operations. This position is in an expanding section of the Laboratories and offers good opportunities for a suitable man. Please write, giving full details and quoting reference 1007, to Central Personnel Services, English Electric Company, Ltd., 24–30, Gillingham Street, London, S.W.1.

**A METALLURGIST** is required for research on the casting and solidification of aluminium and its alloys. An honours degree is essential, and while some research experience would be an advantage, consideration will be given to recent graduates. Applicants should be under 30 years of age. Application should be made to the Director of Research, Aluminium Laboratories, Ltd., Banbury, Oxon.

**A PHYSICAL METALLURGIST** is required for research on aluminium and its alloys, particularly in the textural and alloy metallurgy fields. An honours degree is essential, and while some research experience would be an advantage, consideration will be given to recent graduates. Applicants should be under 30 years of age. Application should be made to the Director of Research, Aluminium Laboratories, Ltd., Banbury, Oxon.

**ANALYTICAL CHEMIST** (Male) with at least 5 years' experience in metallurgical chemical analysis required for analytical laboratory in Research Dept. in North London area. Work involves analysis of ferrous and non-ferrous metals and minerals. Excellent working conditions. Attractive salary according to age and qualifications. Preference given to applicant with University degree. Write Box JIM 713, L.P.E., 110, St. Martin's Lane, London, W.C.2.

**ANALYTICAL RESEARCH CHEMIST** required for Northern Rhodesia.

Applicant must possess good university degree in chemistry or be associate of Royal Institute of Chemistry. One year's experience in chemical laboratory essential, preferably on mixed analytical and research work in inorganic chemistry.

After experience of local conditions he must be capable of conducting experiments without senior supervision. At first applicant would be engaged on semi-routine but varied analytical work, but later, subject to initiative and sound chemical sense, would be expected to undertake work of an applied research character.

Starting basic salary £60 per month plus cost of living allowance, at present about £11 per month. Life Assurance and contributory Pension Scheme. Cash Bonus Scheme, which is at present about 60% on basic salary. Outward passage paid.

Please apply for application form from: Anglo American Corporation of South Africa, Ltd., 11, Old Jewry, London, E.C.2.

**INDUSTRIAL FURNACE MANUFACTURERS** have a vacancy in their Research Department for a young metallurgist with honours degree or equivalent, for work in connection with design and application of heat-treatment and melting furnaces. Previous industrial experience not essential; initiative and ability to deal with a wide variety of problems more important than specialization in one subject; Starting salary in range £500–£650, according to age and experience. good opportunities of advancement for satisfactory candidate. Apply in writing to Chief Metallurgist, Birlec, Ltd., Tyburn Rd., Erdington, Birmingham 24.

**METALLURGISTS** preferably with good honours degrees are required by the Research Laboratories of the General Electric Co., Ltd., Wembley, Middlesex, for experimental research work in the fields of heat-treatment, magnetic materials, and metallography. There is also one vacancy concerned with development work on lamps. Apply to the Staff Manager, stating age, qualifications, and experience.

**NELSON RESEARCH LABORATORIES**, English Electric Company, Ltd., Stafford, have a vacancy in their Metallurgical Laboratory for a senior metallurgist or physicist with metallurgical experience, to lead a team working on the development of magnetic materials. Applicants should have an honours degree and several years' experience in academic or industrial research. This work would have a great appeal for those interested in Physical Metallurgy, and offers ample scope for advancement. Please write, giving full details and quoting reference 310A, to Central Personnel Services, English Electric Company, Ltd., 24–30, Gillingham Street, London, S.W.1.

**YOUNG METALLURGIST** interested in the prospects of future executive position, required by expanding light-engineering and metal-casting company in W. London. Applicants should have L.I.M., H.N.C., or University degree. Starting salary in the region of £550–£650 per annum. Apply: Personnel Manager, Glacier Metal Co., Ltd., Ealing Road, Alperton, Middx.

UNIVERSITY OF BIRMINGHAM

**Department of Industrial Metallurgy**

Applications are invited for the post of Lecturer in Industrial Metallurgy (Grade II) at a salary within the range £550–£1100 p.a.

Applicants must possess a degree in Metallurgy, Engineering, or related science. The Lecturer will be required to deal principally with the Properties and Industrial Uses of Metals, the duties including lecturing and the organization of practical courses. The Lecturer will be encouraged to undertake research, for which facilities are available.

Applications should reach the Registrar, The University, Birmingham, 15, not later than 1 October 1952. Further particulars may be obtained from the undersigned.

C. G. Burton,
*Secretary.*

*The University,*
*Birmingham, 3.*
*August, 1952.*


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